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ARMY ELECTRONICS RESEARCH AND DEVELOPMENT COMMAND FO--ETC F/6 20/2
GROWTH AND CHARACTERIZATION OF BULK, SEMI-INSULATING GALLIUM AR--ETC(U)
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RESEARCH AND DEVELOPMENT TECHNICAL REPORT

DELET-81-12

GROWTH AND CHARACTERIZATION OF BULK, SEMI-
INSULATING GALLIUM ARSENIDE

AD A100634

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MAY 1981

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER DELET-TR-81-12	2. GOVT ACCESSION NO. AD-A100634	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) GROWTH AND CHARACTERIZATION OF BULK, SEMI-INSULATING GALLIUM ARSENIDE.	5. TYPE OF REPORT & PERIOD COVERED	
7. AUTHOR R.L. Ross T.R. AuCoin R.O. Savage J.J. Winter R.O. Malik, R.A. Leupold; C.E. Anderson, Jr., US Army Ballistic Research Laboratory, Aberdeen Proving Ground, MD 21005	6. PERFORMING ORG. REPORT NUMBER	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Electronic Materials Research Division US Army Electronics Technology & Devices Laboratory (ERADCOM) Fort Monmouth, NJ 07703 DELET-ES-S	8. CONTRACT OR GRANT NUMBER(S) 61102A 1L161102AH47 02 051	
11. CONTROLLING OFFICE NAME AND ADDRESS US Army Electronics Research & Development Command Fort Monmouth, NJ 07703 DELET-ES-S	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 12. REPORT DATE May 1981 13. NUMBER OF PAGES 17	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	15. SECURITY CLASS. (of this report) UNCLASSIFIED 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Gallium arsenide, Semi-insulating, Growth, Characterization, Czochralski growth		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The physical and electrical properties of GaAs show it to be an important semiconductor material for use in various electronic devices associated with advanced military systems. However, the realization of enhanced device performance has been delayed, partly due to the lack of consistent, high quality, semi-insulating GaAs substrate material. A modified liquid-encapsulated Czochralski technique employing pressure-assisted, in-situ compounding is described. This process, first demonstrated in the United States by the US Army Electronics Technology and Devices Laboratory, (Cont'd on reverse side)		

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consistently yields high resistivity (to 10^9 ohm-cm) GaAs without the intentional addition of charge compensators. This approach is now becoming the basis for U.S. volume production of large diameter, high quality, semi-insulating GaAs material. An automated system for the measurement of transport properties by use of the van der Pauw method is described. A mixed conduction analysis allows the direct determination of individual carrier concentrations and mobilities, intrinsic carrier concentration and Fermi level. Applied to ET&DL's non-Cr-doped GaAs, this analysis yields electron mobilities higher than Cr-doped material and Fermi levels which are nearly intrinsic.

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SUMMARY

The physical and electrical properties of GaAs show it to be an important semiconductor material for use in advanced military electronic systems. However, millimeter wave and high speed IC device development has been slowed due, in part, to poor and unpredictable quality semi-insulating substrate material. A modification of the liquid encapsulated Czochralski pressure-assisted growth process has been developed which employs in situ compounding of GaAs from its elements. This process, first demonstrated in the U.S. by ET&DL, consistently yields high resistivity (to 10^9 ohm-cm) GaAs without the intentional addition of charge compensators. Transport property determinations by mixed conduction analysis indicate the material has electron mobilities higher than Cr-doped material and Fermi levels which are nearly intrinsic.

The one step technique for "in situ" liquid encapsulated compounding and Czochralski growth of GaAs is believed to be the best approach to solving the current problems associated with semi-insulating substrate fabrication. Various semiconductor material and device manufacturers such as Varian Associates, Westinghouse, Hughes, Rockwell, Microwave Associates and Metals Research have purchased equipment to grow semi-insulating GaAs by this technique⁽¹⁶⁾. Indeed, this approach is now becoming the basis for U.S. volume production of large diameter, high quality, semi-insulating GaAs material.

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INTRODUCTION

A wide variety of semiconductor devices employing gallium arsenide (GaAs) is currently under development by the military for use in advanced communication, surveillance, and target acquisition systems. GaAs, a compound semiconductor exhibiting a large energy bandgap (1.4eV), is characterized by a high intrinsic electron mobility ($8600 \text{ cm}^2 \text{V}^{-1} \text{sec}^{-1}$ @ 300°K) and resistivity ($>10^8 \text{ ohm-cm}$). The low-field electron mobility of GaAs is one of its greatest attributes and offers high frequency operation in devices such as the field effect transistor (FET). Coupled with the material's high peak velocity and low threshold field, GaAs integrated circuit (IC) devices offer a two to six-time speed improvement over their silicon counterparts. When compared with standard silicon IC technology, the process steps for GaAs ICs are relatively simple and few in number. As a result of these advantages, the technology of manufacturing high performance GaAs devices is maturing at a rapid rate. Thus, it is not surprising to find these devices experiencing a greatly expanding role in oscillator, mixer, logic element, power amplification, and low noise/high gain applications. However, the full potential and low-cost manufacture of GaAs devices has yet to be realized, partly due to material problems experienced by substrate suppliers and device manufacturers.

It is commonly held by major GaAs device manufacturers that the quality of semi-insulating substrates is one of the barriers to obtaining reliable, reproducible, high-performance devices. In addition, because the quality of commercially available semi-insulating GaAs substrates is highly variable, extensive material qualification tests are required before substrates are accepted for device fabrication. It has been shown that chromium (Cr), which is used to compensate native impurities and produce high resistivity in GaAs, redistributes during thermal processing(1,2,3), whether during a subsequent epitaxial process or a high temperature ion-implantation anneal procedure. Typically, the Cr depletes from a region one or two microns deep and migrates to the surface where it occupies a region about 400 \AA thick. The most serious consequence of this distribution is that the depletion of Cr can uncompensate some of the native donor impurities existing in the Cr-depleted region, resulting in excess uncontrolled n-type activation and significant changes in device characteristics and reliability. In addition to this "surface conversion" problem, fast diffusing deep-level acceptors, carbon-arsenic vacancy complexes and diffusing deep-level impurities are still observed as a result of process-induced substituents.

In order to minimize the effect of substrate imperfections on devices fabricated by epitaxy, one usually grows a high quality buffer layer to isolate the device from the substrate. However, the epitaxial approach has two major drawbacks: cost, and difficulty in achieving the required thickness and uniformity. Although ion-implantation reduces the cost of active layer fabrication, the important properties of the implanted layers, such as mobility and doping profile, can be significantly affected by substrate characteristics. In addition, inter-device leakage becomes a problem if Cr redistribution causes a thin conducting layer to form on the GaAs surface during the high temperature implant anneal procedure.

The alternative solution to these problems is to improve the quality of substrate material through the reduction of impurity and defect levels and/or the elimination of charge compensators such as Cr. Such is the objective of our research program.

CRYSTAL GROWTH

The technology involved in the growth of GaAs is considerably more complicated than that employed for silicon, for one is dealing with more complicated binary phase equilibria and a highly volatile component - arsenic. Precise control of the As vapor pressure in the chosen growth system is required in order to maintain exact stoichiometry of the GaAs compound during the growth process so as to achieve high mobility and crystal perfection.

The bulk compound is normally formed by the reaction of As vapor with Ga metal at elevated temperatures in sealed quartz ampoules as shown in Figure 1. Typically, an As reservoir contained at one end of the ampoule is heated to 600°C. This generates approximately 1 atm of As vapor pressure in the system, a prerequisite for obtaining stoichiometric GaAs (4). The As vapor reacts with the Ga metal maintained at approximately 1260°C and located at the other end of the ampoule in a quartz boat. After the Ga has been completely reacted, single crystal growth may be initiated by programmed cooling (gradient freeze) or by physically moving either the ampoule or furnace to provide proper temperature gradients for growth (horizontal Bridgeman). Most major commercial GaAs material suppliers to date use either of these processing techniques. Crystals prepared by this technique have a "half moon" or "D" shaped cross-section and, therefore, cause considerable waste in IC processing where the required shape is a circular disk.

Another approach to the commercial production of bulk GaAs which is gaining favor is the liquid encapsulated Czochralski technique (LEC CZ). Metz et al (5) first described the use of an encapsulant to suppress the loss of a volatile component from a melt. Mullin et al (6) used this method for the growth of InAs and GaAs from stoichiometric melts. In this method, shown in Figure 2, the vaporization of As from molten GaAs is inhibited by placing a layer of non-reactive encapsulant (e.g., B₂O₃) on the melt surface. An inert gas pressure which is higher than the As vapor pressure is then maintained over the molten B₂O₃ layer. A rotating seed crystal contacting the molten GaAs is then slowly withdrawn through the liquid encapsulant while the temperature is regulated to obtain the desired diameter crystal. The advantage of the LEC CZ technique is the ability to prepare large diameter crystals in a relatively short period of time. However, the GaAs polycrystalline source material used in this technique, from which the crystal is pulled, is normally compounded in quartz ampoules. The use of quartz as a container for Ga, As, and molten GaAs, or as a reaction vessel, can lead to appreciable silicon contamination (7). Since silicon can act as a shallow donor or acceptor in GaAs (8), chromium, a deep-level acceptor and/or oxygen, a deep-level donor, must be deliberately added in order to compensate the GaAs, thereby making it semi-insulating ($>10^8$ ohm-cm).

A second drawback to the conventional LEC CZ technique is its separate two-step process - compounding and subsequent crystal growth. Because of this increased handling, more impurities can be introduced than those obtained in the horizontal Bridgeman process. The in situ LEC compounding/Czochralski growth process we describe for growing semi-insulating GaAs incorporates both steps and eliminates all dependence on quartz components, thereby reducing or eliminating the requirement for chromium compensation. This is accomplished by reacting elemental gallium and arsenic under a

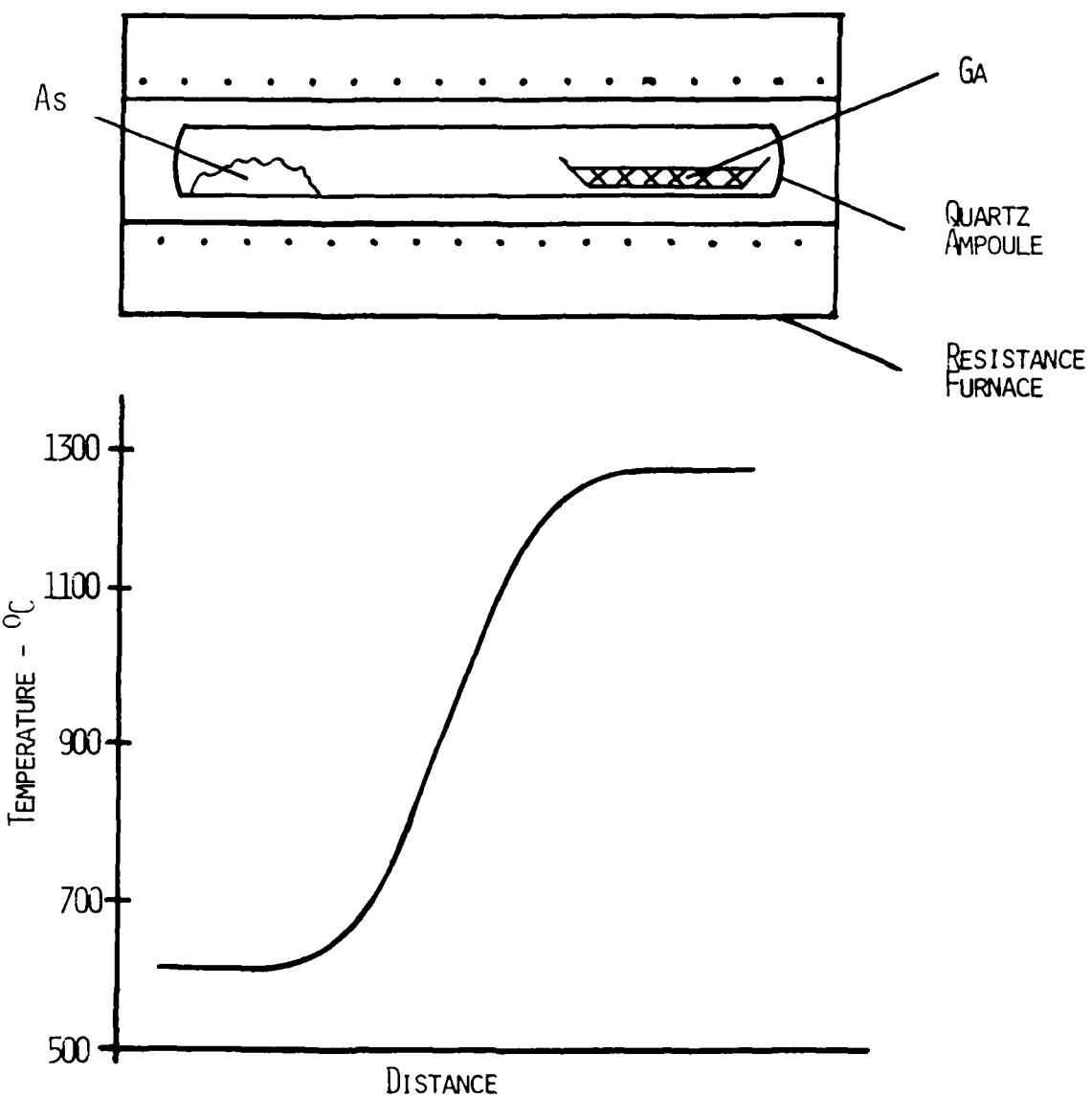


Figure 1. Typical furnace and temperature profile for compounding GaAs in quartz.

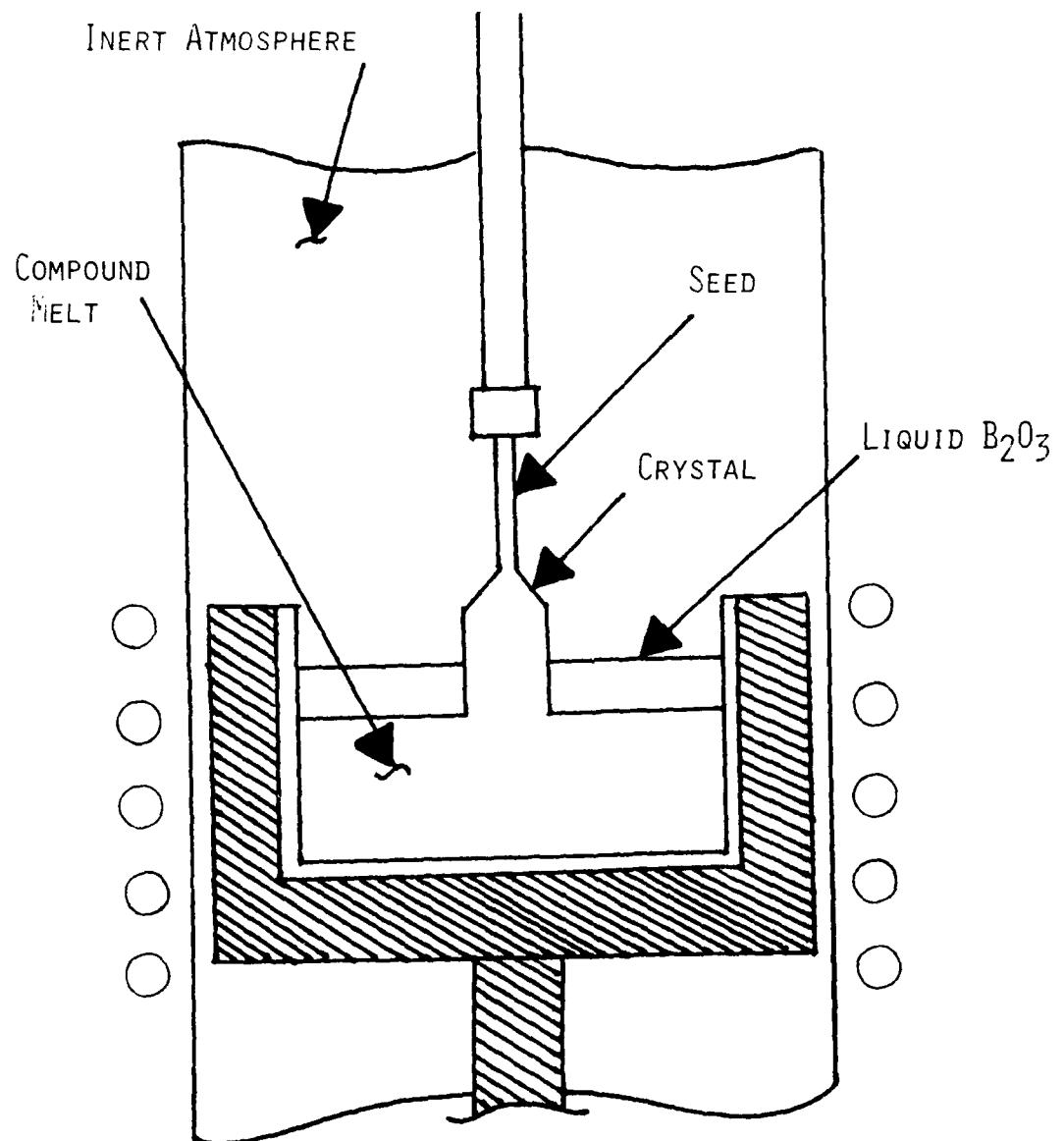


Figure 2. Liquid encapsulated Czochralski pulling technique.

molten encapsulant in pyrolytic boron nitride (PBN) crucibles at nitrogen pressures to 100 atm. A specially designed high pressure Czochralski crystal pulling system, Varian model HPCZ, was used for maintaining the nitrogen gas ambient and for growing the bulk GaAs (see Fig. 3). This system contains provision for the following: rotation of the crystal and/or crucible, raising and lowering of the crystal and/or crucible, remote control of temperature, gas flow, pulling and rotation rates; TV process monitoring, cryo fore-pumping, vac-ion pumping to 10^{-5} torr, and hydraulic lifting of the chamber. It is designed for operation at pressures to 135 atm with neutral, oxidizing or reducing ambients.

The in situ LEC compounding and Czochralski growth of GaAs was carried out in the following manner. The PBN crucibles were etched in HCl, rinsed with de-ionized water followed by methanol, and then vacuum dried overnight at 200°C. The crucibles were then loaded with stoichiometric quantities of high-purity gallium and arsenic. A dehydrated pellet of boron oxide encapsulant was placed on top of the charge and the entire assembly centered within a tantalum or graphite susceptor in the Czochralski pulling chamber as shown in Fig. 4.

It was found that if the boron oxide encapsulant contained an excessive amount of moisture, bubbles continuously formed at the melt/encapsulant interface. These bubbles subsequently rose to the surface releasing arsenic which, in turn, led to nonstoichiometric melts and resultant twinning of the crystals. An oil-free, high-vacuum baking system was constructed to further dehydrate the pellets. Heating the boron oxide to approximately 1000°C at 10^{-7} torr in induction heated platinum/gold crucibles was found to give satisfactory results.

After the growth station was established (susceptor, crucible, gallium, arsenic, boron oxide, etc.) and the chamber closed, the system was evacuated to 10^{-5} torr. The charge was then slowly heated to 325°C to remove residual moisture and volatile gallium/arsenic oxides. If the temperature exceeded 325°C, significant amounts of arsenic were lost by volatilization prior to compounding. After several hours, the system was then backfilled with high-purity nitrogen gas to 3.0 atm and the temperature increased to 450°C. During this step, the boron oxide melted and flowed into the voids. The pressure in the pulling chamber was then increased to 60 atm. As the temperature was slowly increased, the compounding reaction, accompanied by a rapid temperature rise as the crucible approached 700°C, could be observed on the TV monitor.

Single crystals of GaAs were grown from the in situ compounded material by the LEC CZ technique as previously shown in Fig. 2. All crystals were grown in the $\langle 111 \rangle$ -B direction with nitrogen gas pressures of 1.0 to 20.0 atm and pulling rates of 1.0 to 2.0 cm/hr. Both concurrent and countercurrent rotation of crucible and crystal were used at rates of 5 and 15 rpm respectively; the concurrent rotation producing a more convex interface.

ELECTRICAL MEASUREMENTS

The measurements of resistivity, Hall mobility and Hall coefficient were performed using the van der Pauw method⁽⁹⁾. This technique is most advantageous in that only four contacts are required and only the sample thickness need be known. In this work, the conventional "clover-shaped" sample

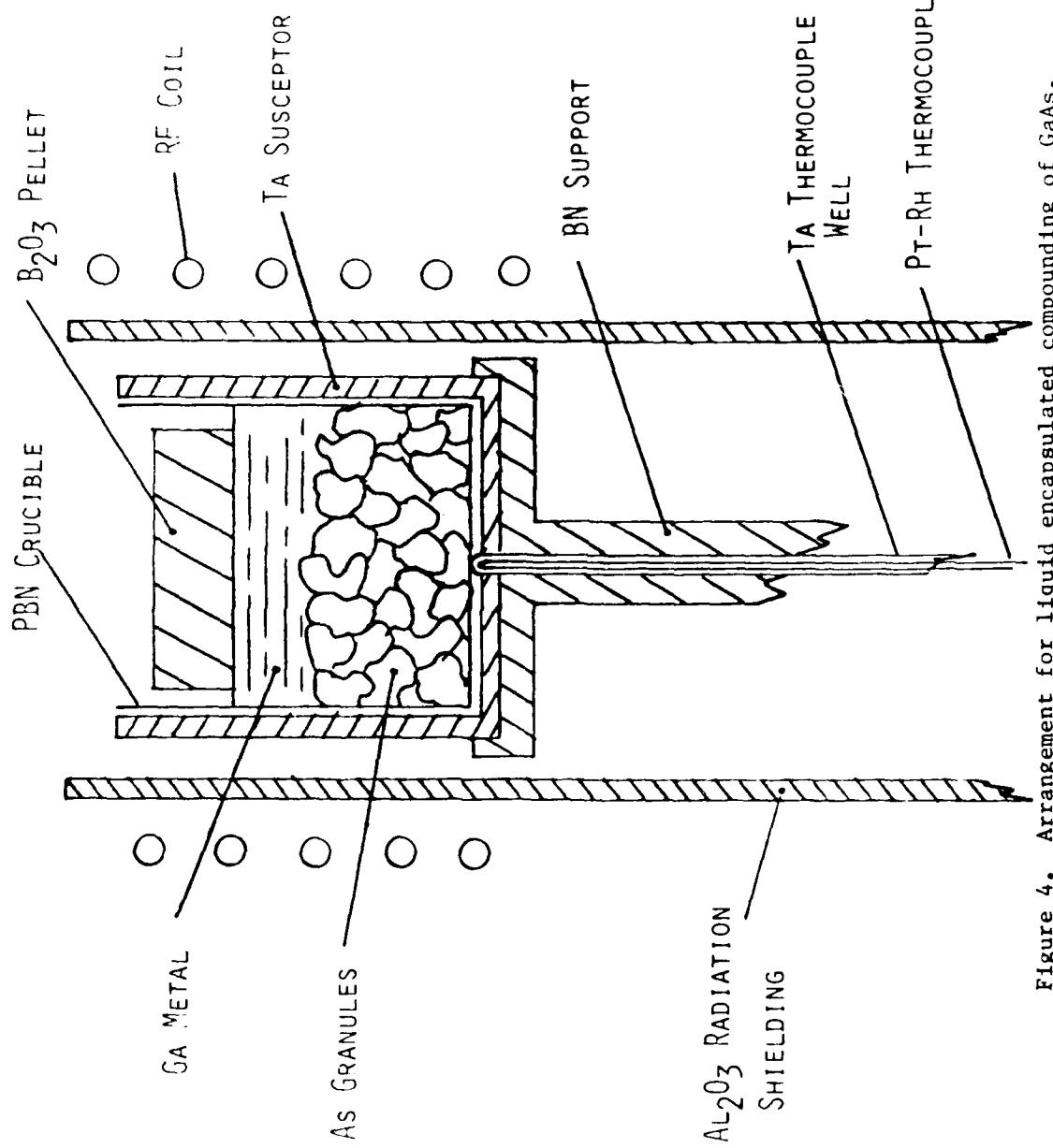
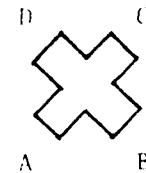


Figure 4. Arrangement for liquid encapsulated compounding of GaAs.

geometry is replaced by an equivalent "Greek cross" structure⁽¹⁰⁾ (see Fig. 5). The actual GaAs structures are produced using an ultra-sonic milling machine.

The resistivity, ρ , in ohm-cm is given by

$$\rho = \frac{\pi t}{\ln 2} \left[\frac{R_{AB,CD} + R_{BC,DA}}{2} \right] F,$$



where $R_{AB,CD} = |V_{CD}| / I_{AB}$

and $R_{BC,DA} = |V_{DA}| / I_{BC}$

Figure 5 Sample geometry.

are in units of ohms, t is the sample thickness in centimeters, and F is the van der Pauw factor, which is a dimensionless quantity dependent only upon the ratio of $R_{AB,CD}/R_{BC,DA}$, and is defined by the transcendental equation

$$\cosh \left[\frac{\ln 2}{F} \cdot \frac{R_{AB,CD}/R_{BC,DA}^{-1}}{R_{AB,CD}/R_{BC,DA}+1} \right] = \frac{1}{2} e^{\ln 2/F} .$$

The Hall coefficient, R_H , in $\text{cm}^3/\text{coulomb}$ is calculated from

$$R_H = \frac{t}{|B|} \Delta R_{AC,BD} \times 10^8$$

where B is the applied magnetic field in gauss and $\Delta R_{AC,BD}$ is the change in resistance when the magnetic field is applied perpendicular to the sample, i.e.,

$$\Delta R_{AC,BD} = \left. \frac{V_{BD}}{I_{AC}} \right|_{B=0} - \left. \frac{V_{BD}}{I_{AC}} \right|_{B=B} ,$$

The Hall mobility, μ_H , in $\text{cm}^2 \text{volt}^{-1} \text{sec}^{-1}$ is given by $\mu_H = |R_H|/\rho$.

The disadvantage of the van der Pauw method is that two resistances ($R_{AB,CD}$ and $R_{BC,DA}$) must be measured, requiring that voltage and current leads be changed. In addition, good experimental practice dictates that leads be reversed and switched in order to compensate for contact resistances and potentials, rectifying effects and thermal emfs. Thus, in the collection of van der Pauw transport data, eight configurations are used for resistivity measurement (Fig. 6 (a)-(h)), and eight for mobility measurement (Fig. 6 (i)-(p)).

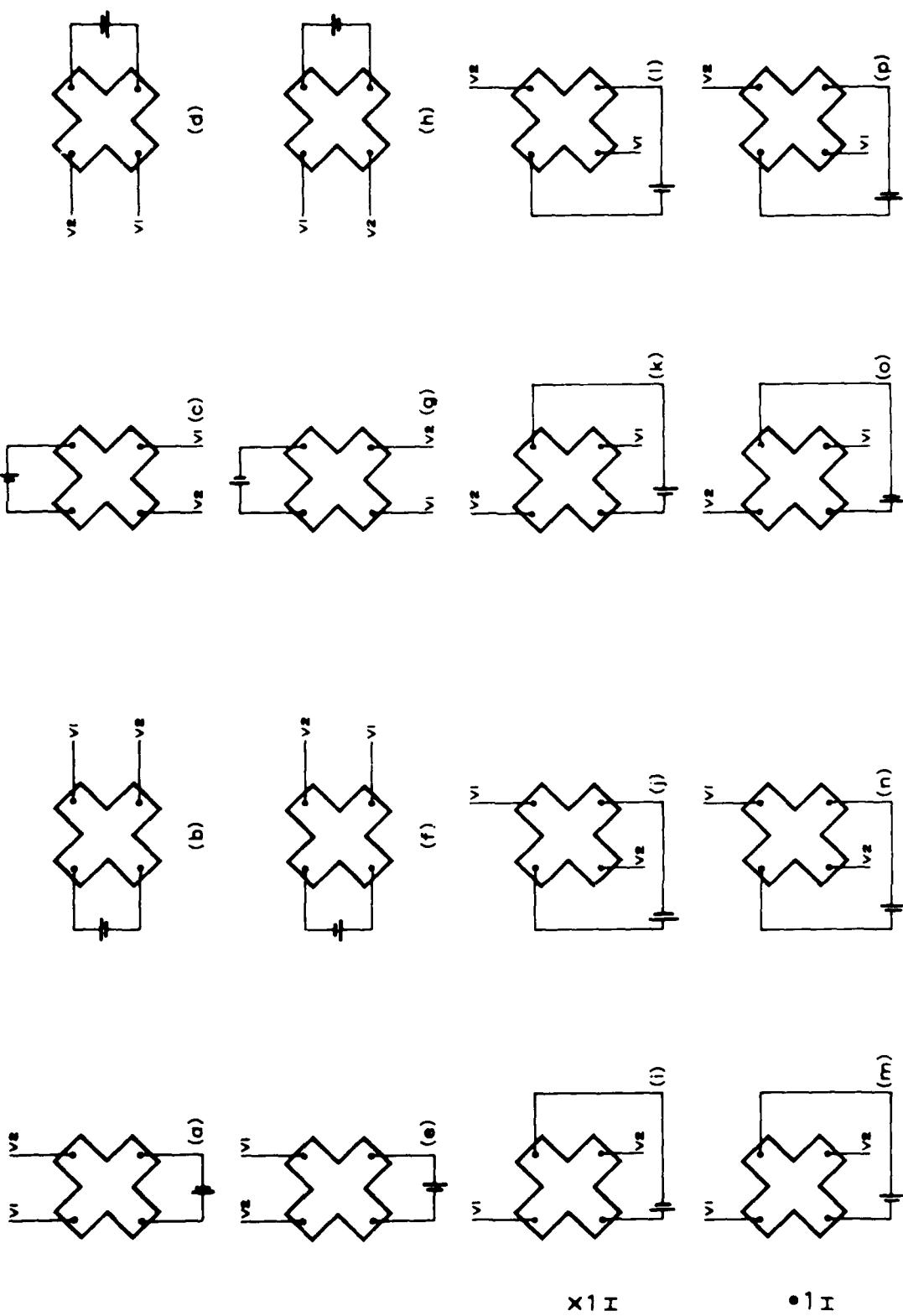


Figure 6. Sample configuration for van der Pauw transport data:
 (a)-(h) resistivity
 (i)-(p) Hall coefficient

The switching of current and voltage leads, and the reversing thereof, are accomplished by the arrangement shown in Fig. 7(11). The sample is contained in a fully shielded and evacuated chamber (Fig. 8) and is connected to four electrometers by triaxial cable, the inner shield of which is driven by the output of each electrometer. Shielded coaxial reed relays provide the required lead switching to generate the various circuit configuration of Fig. 6. The result is a fully guarded system capable of measuring samples having up to 10^{12} ohms resistance with a minimum of noise and settling time.

Because of the large number of measurements necessary and the large number of samples investigated, the guarded van der Pauw system of Fig. 7 was automated whereby all switching of leads and electrometers, measurement of current and voltage, and calculation of parameters are accomplished under the control of a programmable calculator (Fig. 9). The system is presently under expansion to provide for automatic magnetic field selection and temperature control.

MIXED CONDUCTION ANALYSIS

When one carrier type dominates the conduction process, simple Hall coefficient and resistivity measurements at low magnetic fields provide values of carrier concentration and mobility. Semi-insulating GaAs, however, exhibits mixed conduction; i.e., conduction by both holes and electrons. This complication requires additional information - in particular, the magnetic field dependence of both Hall coefficient and resistivity - in order to permit resolution of the independent carrier properties.

The total Hall coefficient is given by

$$R = \frac{R_n \sigma_n^2 + R_p \sigma_p^2 + R_n R_p \sigma_n^2 \sigma_p^2 (R_n + R_p)^2 B^2}{(\sigma_n + \sigma_p)^2 + \sigma_n^2 \sigma_p^2 (R_n + R_p)^2 B^2} \quad (1)$$

and the total conductivity by

$$\sigma = \frac{(\sigma_n + \sigma_p)^2 + \sigma_n^2 \sigma_p^2 (R_n + R_p)^2 B^2}{\sigma_n (1 + R_p^2 \sigma_p^2 B^2) + \sigma_p (1 + R_n^2 \sigma_n^2 B^2)}$$

where the subscripts n and p indicate the contribution due to electrons and holes, respectively, and B is the magnetic field⁽¹²⁾.

Defining the intermediate functions⁽¹³⁾

$$X = \frac{\sigma_n \sigma_p (\sigma_n R_n - \sigma_p R_p)^2}{\mu_n^2 (\sigma_n + \sigma_p)^2}$$

$$Y = \frac{\sigma_n^2 \sigma_p^2 (R_n + R_p)^2}{\mu_n^2 (\sigma_n + \sigma_p)^2}$$

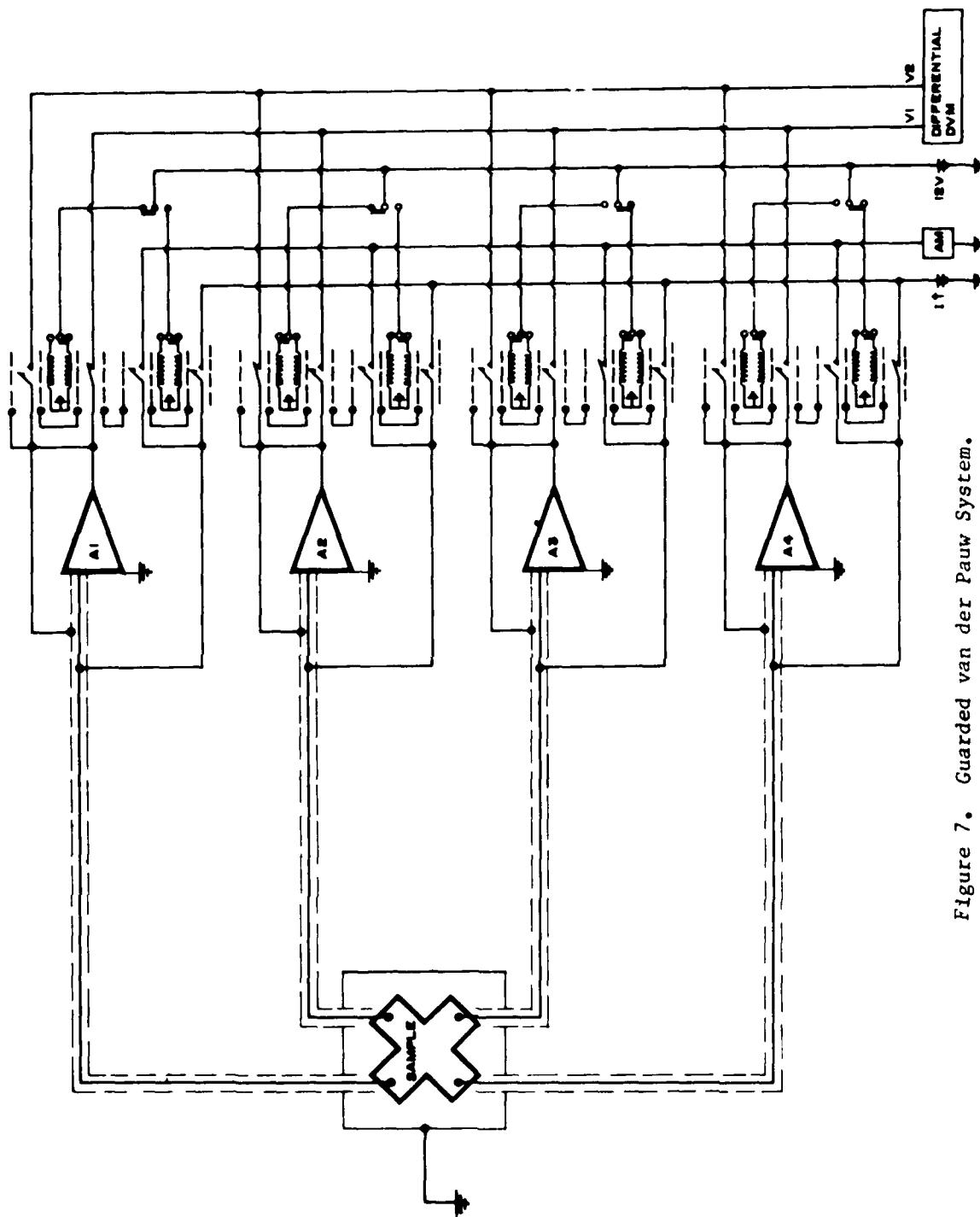


Figure 7. Guarded van der Pauw System.

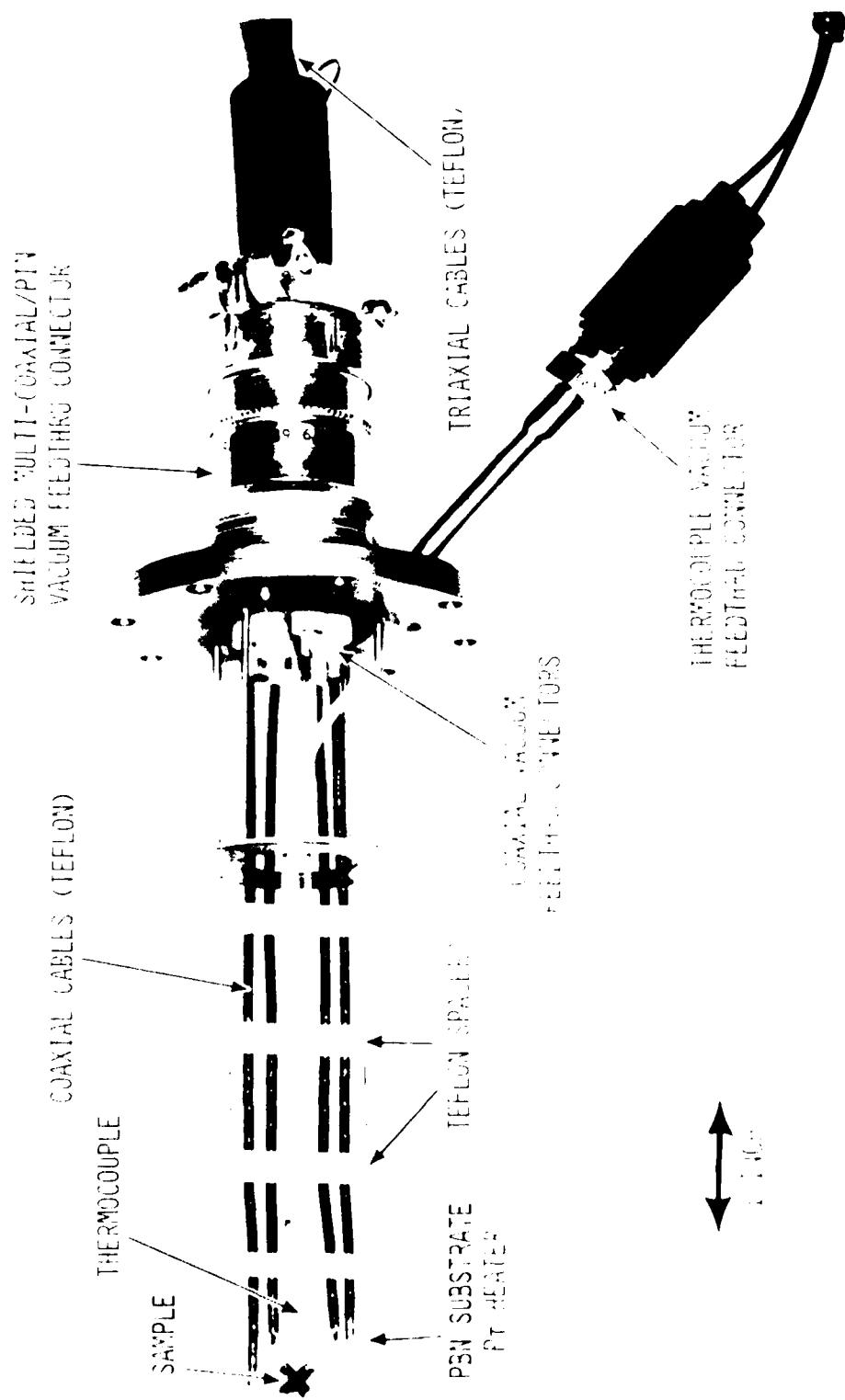


Figure 8. Sample chamber.

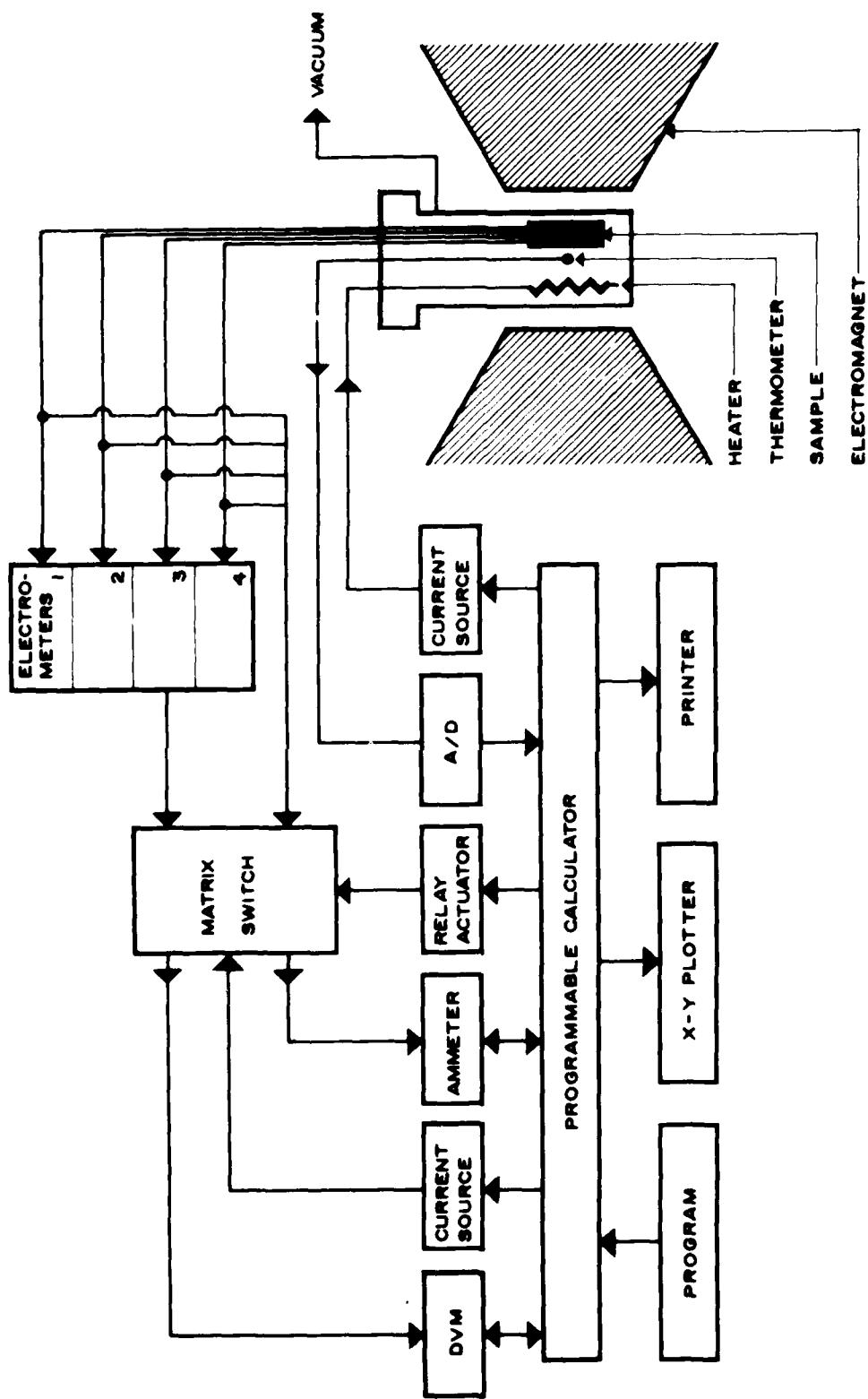


Figure 9. Automated van der Pauw Hall and resistivity system.

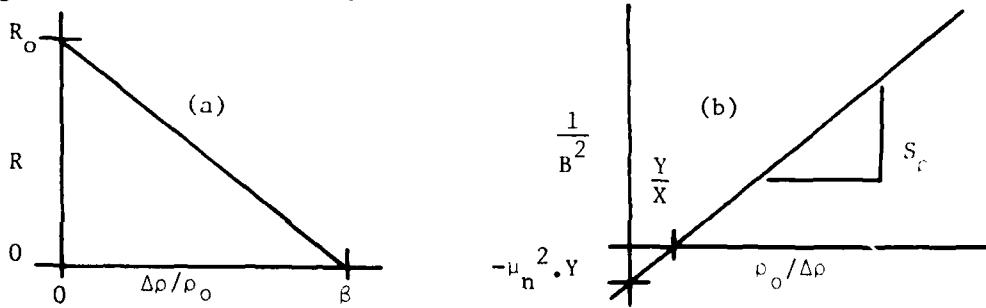
$$Z = - \frac{\sigma_n^2 \sigma_p^2 R_n R_p (R_n + R_p)}{\mu_n^2 (\sigma_n^2 R_n + \sigma_p^2 R_p)}$$

equations (1) and (2) can be rewritten as

$$R = R_0 \left[1 - \frac{(Y + Z)}{X} \frac{\Delta \rho}{\rho_0} \right] \quad (3)$$

$$\frac{1}{B^2} + \mu_n^2 Y = \mu_n^2 X \frac{\rho_0}{\Delta \rho} \quad (4)$$

where ρ_0 is the resistivity at zero magnetic field, $\Delta \rho = \mu(B) - \rho_0$ and is called the magneto-resistivity, and R_0 is the Hall coefficient (equation (1)) at zero magnetic field. Varying the magnetic field from 0 to 18kG and plotting equations (3) and (4) yields curves (a) and (b) as shown.



Curve (a) gives the intercepts R_0 and $\beta = X/(Y+Z)$, whereas curve (b) yields the slope $S_\rho = \mu_n^2 X$, intercept $-\mu_n^2 Y$ and crossover point Y/X .

Letting:

$$T = (R_0 \rho_0)^2 / S_\rho$$

$$A = \frac{2 + T(1 + \beta^2)}{1 - T/\beta}$$

$$b = \frac{1}{2\beta} \left[A + (A^2 - 4)^{1/2} \right]$$

$$c = \frac{1 + \beta b \beta}{b(\beta b + \beta)}$$

where $\iota = r_n/r_p$ and is the ratio of scattering factors and assumed to be unity, then the individual carrier properties can be calculated from:

$$\text{electron mobility} = \mu_n = \frac{1 + \beta^{-1}}{\alpha - b^{-1}} \cdot \frac{(-R_0)\sigma_0}{r_p} \text{ cm}^2 \text{ volt}^{-1} \text{ sec}^{-1},$$

$$\text{hole mobility} = \mu_p = \mu_n/b \text{ cm}^2 \text{ volt}^{-1} \text{ sec}^{-1},$$

$$\text{hole concentration} = p = \frac{\sigma_0}{q\mu_n} \cdot \frac{1}{c + b^{-1}} \text{ cm}^{-3},$$

$$\text{electron concentration} = n = cp \text{ cm}^{-3},$$

$$\text{intrinsic carrier concentration} = n_i = (np)^{1/2} \text{ cm}^{-3}.$$

In addition, the Fermi level can be calculated from

$$E_c - E_F = kT \ln(N_c/n),$$

where N_c is the effective density of states in the conduction band.

An indication of the accuracy of this mixed conduction analysis is given by the linearity of equations (3) and (4) and the similarity of the calculated value of n_i to its theoretical value of $1.5 \times 10^6 \text{ cm}^{-3}$. (13) The analysis ignores the magnetic field dependence of single carrier effects, i.e., it is assumed that $\sigma_{n,p} \neq \sigma_{n,p}(B)$ and $R_{n,p} \neq R_{n,p}(B)$. Recent consideration of single carrier effects indicates they may be of importance and that some modification of the foregoing analysis may be warranted (14, 15).

Table 1 compares the electrical properties of undoped GaAs grown by the ET&DL process to chromium doped substrates from other sources. Although the resistivity is slightly lower, the ET&DL material has one of the highest electron and Hall mobilities, nearly equal carrier concentration and a Fermi energy level nearly intrinsic. Recent growths have resulted in material with resistivities of $0.62 \times 10^9 \text{ ohm-cm}$.

ACKNOWLEDGEMENTS

The authors wish to thank Drs. C. G. Thornton, J. A. Kohn and F. Rothwarf for their technical suggestions, encouragement and support, and Dr. David C. Look of the University of Dayton, Dayton, Ohio, for very helpful discussions.

TABLE 1. COMPARISON OF ELECTRICAL PROPERTIES OF UNDOPED
GaAs (ET&DL) TO CHROMIUM DOPED SUBSTRATES FROM OTHER SOURCES.

ρ_0 $\times 10^9$ ohm cm	$-R_0$ $\times 10^{12}$ cm ² /coul	μ_N $\times 10^3$ cm ² /vsec	μ_P $\times 10^2$ cm ² /vsec	N $\times 10^6$ cm ⁻³	P $\times 10^6$ cm ⁻³	N_I $\times 10^6$ cm ⁻³	$E_C - E_F$ EV	$\mu_H = R_0 \sigma_0$ $\times 10^3$ cm ² /vsec
A2	1.21	2.25	3.41	8.44	.963	2.23	2.76	.684
B2	.697	1.95	5.85	8.42	.832	5.94	2.22	.688
C3	.733	1.80	2.94	4.90	2.48	2.48	2.48	2.80
D1	.0645	.370	5.74	...	16.9660
E1	.843	.647	4.73	5.55	.392	10.0	1.98	2.45
F2	.897	2.48	4.43	5.19	1.04	4.50	2.16	.682
G2	.522	1.55	4.14	3.44	2.14	9.07	4.41	.644
H1	.961	1.14	4.40	4.21	.492	10.2	2.25	.701
ET&DL*	.420	1.67	5.37	9.89	2.16	3.33	2.68	.664
								3.97

*NON-CR DOPED

N_I (THEORETICAL) = 1.5×10^6 cm⁻³

$E_C - E_F$ (THEORETICAL) = .673 EV

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